(19)【発行国】日本国特許庁（ＪＰ）

(12)【公報種別】公開特許公報（Ａ）

(11)【公開番号】特開平５－７８７４３

(43)【公開日】平成５年（１９９３）３月３０日

(54)【発明の名称】磁気特性、被膜特性ともに優れた方向性電磁鋼板の製造方法

(51)【国際特許分類第５版】

C21D 8/12 B 7356-4K

9/46 A 7356-4K

C22C 38/00 303 U 7325-4K

38/18

38/54

C23C 8/26 8116-4K

【審査請求】未請求|

【請求項の数】１

【全頁数】９

(21)【出願番号】特願平３－２４８０９０|

(22)【出願日】平成３年（１９９１）９月２６日

(71)【出願人】

【識別番号】０００００６６５５

【氏名又は名称】新日本製鐵株式会社

【住所又は居所】東京都千代田区大手町２丁目６番３号

(72)【発明者】

【氏名】水口　政義|

(19) [Publication Office] Japanese Patent Office (JP)

(12) [Kind of Document] Japan Unexamined Patent Publication (A)

(11) [Publication Number of Unexamined Application] Japan Unexamined Patent Publication Hei 5 - 78743

(43) [Publication Date of Unexamined Application] 1993 (1993) March 30 days

(54) [Title of Invention] BOTH MAGNETIC PROPERTY AND COATING CHARACTERISTIC IS SUPERIOR MANUFACTURING METHOD OF DIRECTIONALITY ELECTROMAGNETIC STEEL SHEET WHICH

(51) [International Patent Classification 5th Edition]

C21D 8/12 B 7356-4K

9/46 A 7356-4K

C22C 38/00 303 U 7325-4K

38/18

38/54

C23C 8/26 8116-4K

[Request for Examination] Examination not requested

[Number of Claims] 1

[Number of Pages in Document] 9

(21) [Application Number] Japan Patent Application Hei 3 - 248090

(22) [Application Date] 1991 (1991) September 26 day

(71) [Applicant]

[Applicant Code] 000006655

[Name] NIPPON STEEL CORP. (DB 69-057-0072)

[Address] Tokyo Chiyoda-ku Otemachi 2-6-3

(72) [Inventor]

[Name] Water gate Masayoshi

【住所又は居所】福岡県北九州市戸畑区飛幡町１番１号 新日本製鐵株式会社八幡製鐵所内

(72)【発明者】

【氏名】牛神　義行|

【住所又は居所】千葉県富津市新富２０－１　新日本製鐵株式会社技術開発本部内

(74)【代理人】

【弁理士】

【氏名又は名称】大関　和夫

(57)【要約】　　(修正有)

【目的】　方向性電磁鋼板の製造において磁気特性、被膜特性ともに優れた製品を得るための手段を提供する。

【構成】　重量でＣ：０．０２５～０．０９５%、Ｓｉ：２．０～４．５%、Ｓ≦０．０１５%、酸可溶性Ａｌ：０．０１０～０．０６０%、Ｎ：０．００１０～０．０１３０%、Ｍｎ：０．０５０～０．４５%、Ｃｒ：０．０４～０．２５%、Ｐ：０．００５～０．０４５%を含み、更に微量添加元素として、Ｐｂ、Ｔｅ、Ｍｏ、Ｎｂ、Ｓｅ、Ｎｉ、Ｖ、Ｓｂ、Ｂ、Ｔｉ、Ｓｎのいずれかの元素の１種類以上を添加量として０．００５～０．３%含み、残部Ｆｅ及び不可避的不純物からなる電磁鋼スラブを、１２８０℃未満の温度に加熱し、熱間圧延し、熱延まま又は熱延板焼鈍し、１回又は中間焼鈍を挟んで２回以上冷間圧延し、次いで脱炭焼鈍した後、仕上焼鈍する方向性電磁鋼板の製造プロセスにおいて、脱炭焼鈍後、ストリップを走行せしめる状態で、アンモニアを含むガス中で窒化処理を行うことを特徴とする。

[Address] Inside of Fukuoka Prefecture Kitakyushu City Tobata-ku flying Hata Cho 1-1　Nippon Steel Corp. (DB 69-057-0072) Yawata mak鐵 place

(72) [Inventor]

[Name] Cattle God　Yoshiyuki

[Address] Inside of Chiba Prefecture Futtsu City Shintomi 20 - 1 Nippon Steel Corp. (DB 69-057-0072) technological development headquarters

(74) [Attorney(s) Representing All Applicants]

[Patent Attorney]

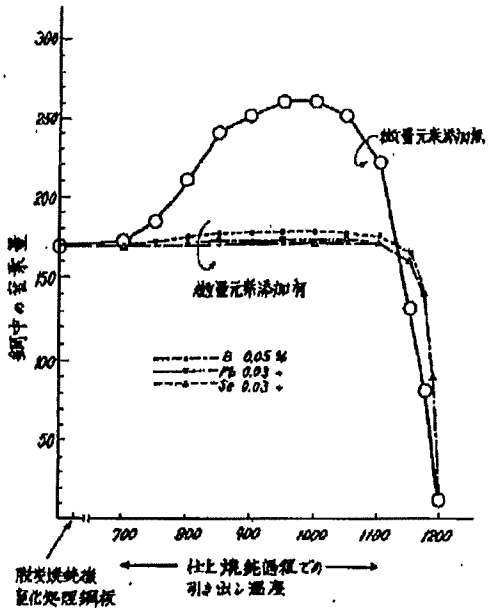
[Name] OZEKI　KAZUO

(57) [Abstract]　　(There is an amendment.)

[Objective] Both magnetic property and coating characteristi c means in order to obtain productwhich is superior is offered at time of producing directionality electromagnetic steel sheet.

[Constitution] With weight C: 0.025 to 0.095 %, Si:2.0 to 4.5 %, S 0.015 %, acid soluble Al :0.010 to 0.060 %, N:0. 0010 to 0.0130 %, Mn :0.050 to 0.45 %, Cr :0.04 to 0.25 %, P:0.005 to 0.045 % including, Furthermore trace addition element doing, Pb, Te, Mo, Nb, Se, Ni, V, Sb, B, Ti, With 1 kind or more of element of any of Sn as addition quantity the0. 005 to 0.3 % including, Consists of remainder Fe and unavoidable impurity electromagnetic steel slub which, It heats to temperature under 1280 °C, hot rolling does, hot rolling wayor hot rolled plate annealing does, puts between one time or intermediate annealing and cold rollingabove twice does, after decarbonizing annealing doing next, after decarbonizing annealing, the strip with state which runs, it designates that nitriding is donein gas which includes ammonia as feature in production process of thedirectionality electromagnetic steel

plate which finished annealing is done.



【特許請求の範囲】

【請求項１】　重量でＣ：０．０２５～０．０９５%、Ｓｉ：２．０～４．５%、Ｓ≦０．０１５%、酸可溶性Ａｌ：０．０１０～０．０６０%、Ｎ：０．００１０～０．０１３０%、Ｍｎ：０．０５０～０．４５%、Ｃｒ：０．０４～０．２５%、Ｐ：０．００５～０．０４５%を含み、更に微量添加元素として、Ｐｂ、Ｔｅ、Ｍｏ、Ｎｂ、Ｓｅ、Ｎｉ、Ｖ、Ｓｂ、Ｂ、Ｔｉ、Ｓｎのいずれかの元素の１種類以上を添加量として０．００５～０．３%含み、残部Ｆｅ及び不可避的不純物からなる電磁鋼スラブを、１２８０℃未満の温度に加熱し、熱間圧延し、熱延まま又は熱延板焼鈍し、１回又は中間焼鈍を挟んで２回以上冷間圧延し、次いで脱炭焼鈍した後、仕上焼鈍する方向性電磁鋼板の製造プロセスにおいて、脱炭焼鈍後、ストリップを走行せしめる状態で、アンモニアを含むガス中で窒化処理を行うことを特徴とする磁気特性、被膜特性ともに優れた方向性電磁鋼板の製造方法。

[Claim(s)]

[Claim 1] With weight C: 0.025 to 0.095 %, Si:2.0 to 4.5 %, S 0.015 %, acid soluble Al :0.010 to 0.060 %, N:0.0010 to 0.0130 %, Mn :0.050 to 0.45 %, Cr :0.04 to 0.25 %, P:0.005 to 0.045 % including, Furthermore trace addition element doing, Pb, Te, Mo, Nb, Se, Ni, V, Sb, B, Ti, With 1 kind or more of element of any of Sn as addition quantity the0.005 to 0.3 % including, Consists of remainder Fe and unavoidable impurity electromagnetic steel slub which, It heats to temperature under 1280 °C, After hot rolling it does, hot rolling way or hot rolled plate annealing does, putsbetween one time or intermediate annealing and cold rolling above twice does, thedecarbonizing annealing doing next, in production process of directionality electromagnetic steel sheet which finished annealingis done, after decarbonizing annealing, strip with state which runs, boththe magnetic property and coating characteristic which designate that nitriding is done in thegas which includes ammonia as feature manufacturing method of directionality electromagnetic steel sheet whichis superior.

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は、電気機器の鉄心材料と

[Description of the Invention]

[0001]

[Field of Industrial Application] As for this invention, it regards

して用いられる方向性電磁鋼板の製造方法に関し、磁気特性、被膜特性ともに優れた方向性電磁鋼板の製造を可能にするものである。

manufacturing method of directionality electromagnetic steel sheet which is used asthe iron core material of electric equipment, both magnetic property and coating characteristic it is somethingwhich makes production of directionality electromagnetic steel sheet which is superior possible.

【０００２】

[0002]

【従来の技術】方向性電磁鋼板は主としてトランス、発電機、その他の電気機器の鉄心材料として用いられ、磁気特性、特に鉄損特性が良好でなければならない。方向性電磁鋼板は二次再結晶現象を利用して圧延面に（１１０）面、圧延方向に〔００１〕軸をもった、いわゆるゴス方位を有する結晶粒を発達させることにより得られる。

[Prior Art] Directionality electromagnetic steel plate is used and as iron core material of transformer , electric generator and theother electric equipment mainly magnetic property and especially iron loss characteristic must besatisfactory. crystal grain where directionality electromagnetic steel plate on rolling aspect (110) plane , (001)had axis in rolling direction making use of secondary recrystallization phenomenon, possesses the so-calledゴス orientation is acquired by advancing.

【０００３】二次再結晶は周知のように仕上焼鈍で生じるが、二次再結晶温度域まで一次再結晶の成長を抑制する微細なＡｌＮ、ＭｎＳｅ等の析出物、いわゆるインヒビターを存在させる必要がある。このため、電磁鋼スラブは、例えば１３５０～１４００℃程度の高温度に加熱され、インヒビターを形成する成分、例えばＡｌ、Ｍｎ、Ｓ、Ｓｅ、Ｎ等を完全に固溶させ、熱延板あるいは最終冷延前の中間板においてインヒビターを微細に析出させる焼鈍が行われている。

[0003] Widely known way it finishes secondary recrystallizatio n and occurs with annealing,but microscopic AlN , MnS e or other precipitate and so-called inhibitor which control growth of theprimary recrystallization it is necessary to exist to secondary recrystallization temperature region. Because of this, electromagnetic steel slab is heated by high temperature of for example 1350 to 1400 °C extent, thesolid solution doing component , for example Al , Mn , S, Se andthe N etc which form inhibitor completely, annealing whichprecipitates inhibitor to fine in intermediate plate before hot rolled plate or finalrolling is done.

【０００４】かかる処理を施すことにより磁束密度の高い方向性電磁鋼板が製造されるようになったが、電磁鋼スラブの加熱は前述のように高温で行われるために、溶融スケールの発生量が大で加熱炉の操業に支障をきたす。また加熱炉のエネルギー原単位高や表面疵の発生等の問題がある。スラブ加熱温度を下げた方向性電磁鋼板製造法が検討されている。例えば、特開昭５２－２４１１６号公報ではＡｌの他にＺｒ、Ｔｉ、Ｂ、Ｎｂ、Ｔａ、Ｖ、Ｃｒ、Ｍｏ等の窒化物形成元素を含有させることにより、スラブ加熱を１１００～１２６０℃で行う製造法が開示されている。また、特開昭５９－５６５２２号公報ではＭｎを０．０８～０．４５%、Ｓを０．００７%以下とし、〔Ｍｎ〕×〔Ｓ〕積を下げ、さらにＡｌ、Ｐ、Ｎを含有させた電磁鋼スラブを素材とする製造法を提案している。

[0004] It reached point where directionality electromagnetic st eel plate where magnetic flux density is high byadministering this treatment is produced, but heating electromagnetic steel slab theaforementioned way because it is done with high temperature, generated amount of the dissolving scale being large, causes hindrance to operation ofthe furnace. In addition energy source unit of furnace high and there is an occurrenceor other problem of surface scratch. directionality electromagnetic steel sheet structure method which lowered slab heating temperature is examined. production method which heats slab with for example Japan Unexamined Patent Publication Showa 52 - 24116 disclosure by containing Zr,the Ti, B, Nb, Ta, V, Cr andthe Mo or other nitride formation element to other than Al, with 110 0 to 1260 °C isdisclosed. In addition, with Japan Unexamined Patent Publication Showa 59 - 56522 disclosure Mn 0.08 to 0.45 % and S aredesignated as 0.007 % or lower, (Mn) X (S) product is lowered, furthermore production method which designates electromagnetic steel slab which contains theAl , P and N as material is proposed.

【０００５】低温スラブ加熱方法は一定の作用効果が奏されているが、インヒビター形成成分、例えばＡｌ、Ｍｎ、Ｓ、Ｓｅ、Ｎ等が鋼中に完全に固溶されていないか

[0005] Low temperature slub heating method has had fixed acti ng effect , but because inhibitor formationcomponent , for example Al , Mn , S, Se and N etcthe solid solution are not

ら、二次再結晶の発現に効果的なインヒビターを形成することが課題である。本出願人は特開昭６３－１００１１１号で脱炭焼鈍時に所定板厚に冷間圧延された方向性電磁鋼板をストリップ状で通板する際にNH_3を用いて窒化させ、インヒビターを作り込む製造方法を提案した。

done completely in steel, fact that theeffective inhibitor is formed in revelation of secondary recrystallization is problem. This applicant when with Japan Unexamined Patent Publication Showa 63 - 100111 number at time of decarbonizing annealing in thespecified plate thickness sheet passage doing directionality electromagnetic steel sheet which cold rolling is done with thestrip, nitriding doing making use of NH3 , made inhibitor andproposed manufacturing method which is packed.

【０００６】

【発明が解決しようとする課題】NH_3等により〔Ａｌ、Ｓｉ〕Ｎを主成分とするインヒビターを形成させた場合、仕上焼鈍時に雰囲気ガス中のN_2の鋼板への吸収（窒化）又は雰囲気ガス中へ鋼板からN_2の脱離が生じ、インヒビターの強度が一定とならず、結果として二次再結晶の発現が不安定となる場合がある。これらの現象は、鋼板の板厚が薄くなるほど反応の界面積が増大し、仕上焼鈍時に雰囲気の影響を強く受けるためにインヒビターの強度も変わり、二次再結晶発現の不安定性が一層助長されるためと考えられる。その結果、薄手材ほど磁性の再現性が乏しく、又磁気特性もあまりよくなかった。

[0006]

[Problems to be Solved by the Invention] When (Al and Si) in hibitor which designates N as themain component was formed with NH3 etc, at time of finished annealingto absorption (nitriding) to steel plate of N2 in atmosphere gas orin atmosphere gas removal of N2 occurs from steel plate, strengthof inhibitor does not become fixed, there are times when revelation ofthe secondary recrystallization becomes unstable as result. interfacial area of extent reaction where plate thickness of steel plate becomesthin increases these phenomenon, finishes and also strength of inhibitorchanges in order to receive influence of atmosphere strongly at thetime of annealing, is thought for sake of instability of secondary recrystallizationrevelation is more promoted. As a result, about light material reproducibility of magnetism to bescanty, in addition either magnetic property without being good excessively.

【０００７】又、仕上焼鈍において被膜形成後、二次再結晶が発現するが、元々二次再結晶は、鋼板中の〔Ａｌ、Ｓｉ〕と結びついた窒素が脱離することにより生じるものであり、脱離後の窒素は被膜を介して鋼板から雰囲気ガス中に放出される。仕上焼鈍時に鋼板が過剰の窒素を吸収すると、二次再結晶時に被膜をつき破って鋼板から窒素が放出され、いわゆるシモフリと呼ばれる地鉄の露出した被膜不良部が発生する。しかも、鋼板の板厚が薄くなるほど反応の界面積が増大し、仕上焼鈍時に多量の窒素を吸収するために二次再結晶後の被膜を介した放出窒素量も増加し、シモフリは発生し易い。

[0007] After coating formation, secondary recrystallization re veals in also, finished annealing, butthe originally secondary recrystallization is something which it occurs nitrogenwhich is related with in steel sheet (Al and Si) by liberationdoing, nitrogen after liberation through coating, from steel sheet isdischarged in atmosphere gas. When steel sheet absorbs nitrogen of excess at time of thefinished annealing, it is attached at time of secondary recrystallization and tears thecoating and nitrogen is discharged from steel sheet, coating defective part which thearea iron which is called so-callシモフ jp9 exposes occurs. Furthermore, interfacial area of extent reaction where plate thickness of the steel sheet becomes thin increases, finishes and is through coating afterthe secondary recrystallization in order to absorb nitrogen of large amount at time of theannealing also discharge nitrogen amount to increase, シモフ jp9 is easyto occur.

【０００８】本発明者らは、窒化物のみをインヒビターとする方向性電磁鋼板の磁性と被膜の安定化は、仕上焼鈍時の鋼板の窒化、および窒素の抜け等インヒビターの弱体化をいかに防ぐかにあると考えた。そこで、仕上焼鈍時の鋼板の窒素の吸収、放出現象を明らかにするために、様々な元素を製鋼段階で添加し、最初に脱炭焼鈍後の鋼板の窒化特性を調べた。

[0008] That it is, you thought magnetism of directionality elect romagnetic steel sheet which designates onlythe nitride as inhibitor and stabilization of coating, it finishes thethese inventors and, how prevents body weakening of inhibitor such as nitriding of thesteel sheet at time of annealing, and coming out of nitrogen. It finished then and, in order to make absorption and release phenomenon ofthe nitrogen of steel

sheet at time of annealing clear, it added thevarious element with steelmaking step, inspected nitriding characteristic of steel sheet afterthe decarbonizing annealing first.

【０００９】その結果、Ｐｂ、Ｓｅ、Ｓｂ、Ｂ等の粒界偏析型の成分を添加した脱炭焼鈍時は、無添加材に比し、窒化が生じにくいことが判明した。この原因としては、これらの元素を添加した材料は脱炭焼鈍後バリヤーを形成し、窒化を阻害しているものと推定している。さらに、これらの元素を添加した脱炭焼鈍板をNH_3濃度を高くして、所定量の窒素を確保した後、仕上焼鈍を行い、焼鈍過程での鋼板の窒化量を調べたところ、無添加材に比し、仕上焼鈍過程で鋼板の窒素量が殆ど増加しないことが判った。又、その時、同時に仕上焼鈍過程における一次再結晶の大きさを調べてみると、微量元素を添加した鋼板は、無添加材に比し、高温領域まで脱炭焼鈍後の一次再結晶粒径が保持されていることが判った。

[0009] As a result, it compared time of decarbonizing annealing which adds component ofthe Pb , Se, Sb and B or other grain boundary segregation type, to no addition material,the nitriding is difficult occurring was ascertained. As this cause, material which adds these element forms barrier afterthe decarbonizing annealing, those which obstruct nitriding has presumed. Furthermore, making NH3 concentration high, after guaranteeing nitrogen ofthe predetermined amount, it did finished annealing, when nitriding quantity of thesteel sheet with annealing process was inspected, it compared decarbonizing annealing sheet which addsthese element to no addition material, finished and it understood that thenitrogen amount of steel sheet almost does not increase with annealing process. When that time of also,, it finishes simultaneously and it triesinspecting size of primary recrystallization in annealing process, it compared steel sheetwhich adds trace elements, to no addition material, it understood to the high temperature region that primary recrystallization particle diameter after decarbonizing annealing is kept.

【００１０】即ち、これらの微量の元素を添加した鋼板は、仕上焼鈍過程での窒化を阻害すると同時に、一次再結晶粒の成長抑制効果もあった。従来、窒化物のみをインヒビターとする方向性電磁鋼板の弱点であった薄手材ほどシモフリの発生頻度が高く、かつ磁性が不安定となる現象がＰｂ、Ｓｅ、Ｓｂ、Ｂ等の粒界面偏析型元素を添加することにより解消した。|

[0010] Namely, when it finishes steel plate which adds element of these trace amountand, obstructs nitriding with annealing process simultaneously, there was alsoa growth supression effect of primary recrystallization grain. Until recently, about light material which is a weak point of directionality electromagnetic steel platewhich designates only nitride as inhibitor frequency of occurrence ofシモフjp9 is high, it cancelled due to fact that phenomenon where atthe same time magnetism becomes unstable adds Pb , Se,the Sb and B or other grain boundary surface segregation type element.

【００１１】このように本発明はシモフリがなく、グラスが良好でかつ二次再結晶も安定して発現し、磁気特性的にも良好な方向性電磁鋼板をストリップの窒化法で得ることができる。

[0011] This way this invention not to beシモフjp9, glass be ingsatisfactory and and also secondary recrystallization stabilizing, it can reveal, canacquire satisfactory directionality electromagnetic steel sheet even magnetic property with nitriding method of strip.

【００１２】

[0012]

【課題を解決するための手段】本発明の要旨とするところは、重量でＣ：０．０２５～０．０９５%、Ｓｉ：２．０～４．５%、Ｓ≦０．０１５%、酸可溶性Ａｌ：０．０１０～０．０６０%、Ｎ：０．００１０～０．０１３０%、Ｍｎ：０．０５０～０．４５%、Ｃｒ：０．０４～０．２５%、Ｐ：０．００５～０．０４５%を含み、更に微量添加元素として、Ｐｂ、Ｔｅ、Ｍｏ、Ｎｂ、Ｓｅ、Ｎｉ、Ｖ、Ｓｂ、Ｂ、Ｔｉ、Ｓｎのいずれかの元素の１種類以上を添加量として０．００５～０．３%含み、残部Ｆｅ及び不可避的不純物からなる電磁鋼スラブ

[Means to Solve the Problems] As for gist of this invention, Wi th weight C:0.025 to 0.095 %, Si:2.0 to 4.5 %, S 0.015 %, acid soluble Al :0.010 to 0.060 %, N:0.0010 to 0.0130 %, Mn :0.050 to 0.45 %, Cr :0.04 to 0.25 %, P:0.005 to 0.045 % including, Furthermore trace addition element doing, Pb , Te , Mo, Nb, Se, Ni , V, Sb , B, Ti, With 1 kind or more of element of any of Sn as addition quantity the0.005 to 0.3 % including, Consists of remainder Fe and unavoidable impurity electromagnetic steel slub which, It heats to temperature under 1280 °C, After hot rolling it does, hot rolling way or hot

を、１２８０℃未満の温度に加熱し、熱間圧延し、熱延まま又は熱延板焼鈍し、１回又は中間焼鈍を挟んで２回以上冷間圧延し、次いで脱炭焼鈍した後、仕上焼鈍する方向性電磁鋼板の製造プロセスにおいて、脱炭焼鈍後、ストリップを走行せしめる状態で、アンモニアを含むガス中で窒化処理を行うことを特徴とする磁気特性、被膜特性ともに優れた方向性電磁鋼板の製造方法にある。

rolled plate annealing does, putsbetween one time or intermediate annealing and cold rolling above twice does, thedecarbonizing annealing doing next, after decarbonizing annealing, strip with state whichruns, both magnetic property and coating characteristic which designate that nitriding isdone in gas which includes ammonia as feature there is a manufacturing methodof directionality electromagnetic steel sheet which is superior in production process of directionality electromagnetic steel sheet which thefinished annealing is done.

【００１３】以下、本発明について詳細に説明する。本発明者等は、電磁鋼スラブを１２８０℃未満の温度で加熱する低温スラブ加熱を適用して磁気特性及び被膜外観性の優れた方向性電磁鋼板を安定して製造すべく検討した。その結果、鋼中に、Ｐｂ、Ｔｅ、Ｍｏ、Ｎｂ、Ｓｅ、Ｎｉ、Ｖ、Ｓｂ、Ｔｉ、Ｓｎ等のいずれかの元素の１種類以上を微量添加すると、ストリップ窒化でインヒビターを形成させる方向性電磁鋼板において磁気特性が優れ、かつ被膜が良好なものが得られることを見出した。

[0013] You explain in detail below, concerning this invention. Applying low temperature slub heating which heats electromagnetic steel slub with temperature under the1280 °C, stabilizing directionality electromagnetic steel sheet where magnetic property and coating external appearance aresuperior, in order that it produces, it examined this inventor etc. As a result, when in steel, 1 kind or more of element of Pb ,the Te , Mo, Nb, Se, Ni , V, the Sb , Ti and Sn or other any trace addition is done, magnetic property issuperior in directionality electromagnetic steel sheet which forms inhibitor with strip nitriding, factthat those where at same time coating is satisfactory areacquired was discovered.

【００１４】本発明が適用される電磁鋼スラブの成分組成は次のとおりである。Ｃの含有量が少なくなると二次再結晶が不安定となるので０．０２５%以上とする。一方、その含有量が多くなりすぎると脱炭焼鈍時間が長くなるので０．０９５%以下とする。Ｓｉは鉄損の低下、インヒビター形成のために必要な成分で、そのために２．０%以上含有させる。一方、その含有量が多くなると冷間圧延時に割れ発生が多発するので４．５%以下とする。

[0014] Component composition of electromagnetic steel slab w here this invention is applied is as follows. When content of C decreases, because secondary recrystallization becomes theunstable, it makes 0.025 % or higher. On one hand, when content becomes too many, because decarbonizing annealing timebecomes long, it makes 0.095 % or lower. Si with component which is necessary for decreasing and inhibitorformation iron loss, 2.0 % or higher contains because of that. On one hand, when content becomes many, because split occursfrequently at time of cold rolling, it makes 4.5 % or lower.

【００１５】Ｍｎは熱間脆性を防ぐとともに、グラス被膜を良質化する作用があり、これを奏するには０．０５０%以上必要である。一方、その含有量が増えると、磁束密度が劣化するので０．４５%以下とする。さらに本発明ではスラブ加熱を１２８０℃未満で行うので、例えばＳとの化合物であるＭｎＳは完全固溶せず、インヒビターとしてＭｎＳを用いない点からもその上限は前述のとおりとする。

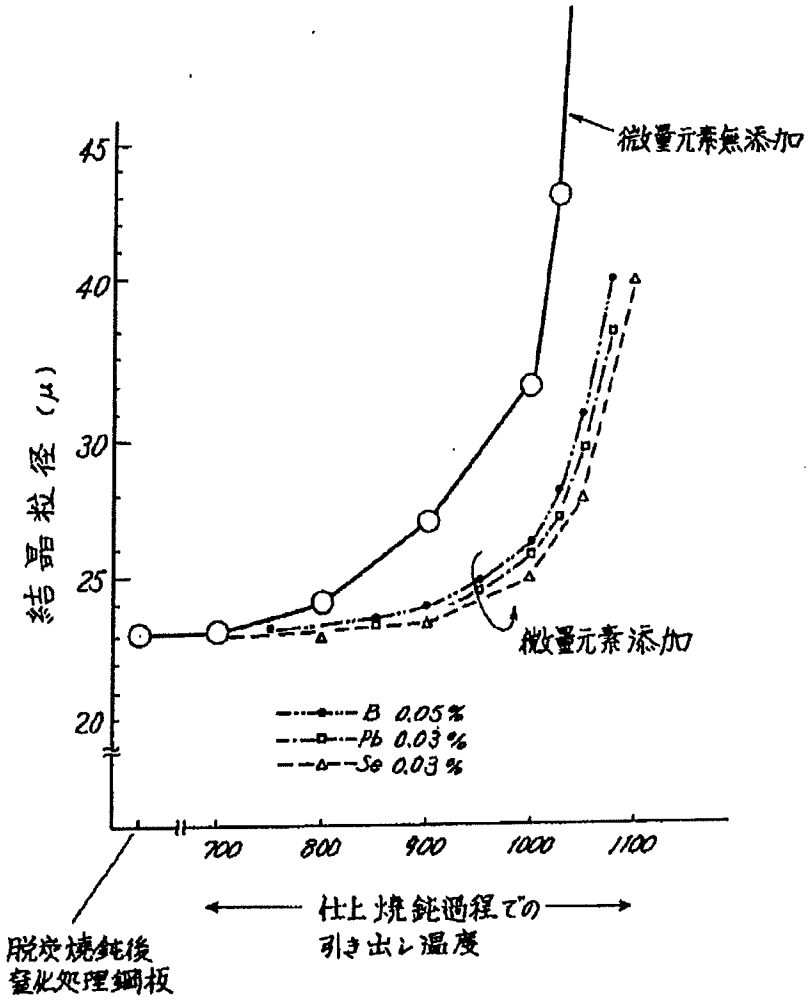
[0015] As for Mn as high-temperature brittle is prevented, for t hereto be action which glass coating to good quality is converted, to possessthis it is 0.050 % or higher necessary. On one hand, when content increases, because magnetic flux density deteriorates,it makes 0.45 % or lower. Furthermore because with this invention it heats slab under 1280 °C, theMnS which is a compound of for example S does not do complete solid solution,the upper limit makes aforementioned sort even from point which does notuse MnS as inhibitor.

【００１６】Ｓは偏析を生じやすく、正常な二次再結晶粒の成長を妨げる原因となるために０．０１５%以下とする。ＡｌはＮあるいはＳｉと結合して（Ａｌ、Ｓｉ）Ｎを形成し、二次再結晶の安定化に必要であり、そのために０．０１０%以上含有させる。一方、本発明でのスラブ加熱温度は１２８０℃未満でＡｌを不完全に固溶させることから、ＡｌＮの含有量が多くなると熱間圧延の

[0016] It makes 0.015 % or lower because it becomes cause whe re S iseasy to cause segregation, growth of normal secondary recrystallization grain obstructs. Al connecting with N or Si, forms (Al and Si) N,it is necessary for stabilization of secondary recrystallization, 0.010 % or higher itcontains because of that. On one hand, because as for slab heating temperature with this invention when Albecomes imperfectly

【図２】

[Figure 2]


45
40
30
25
20
結晶粒径（μ）
微量元素無添加
微量元素添加
B 0.05%
Pb 0.03%
Se 0.03%
700
800
900
1000
1100
仕上焼鈍過程での
引き出し温度
脱炭焼鈍後
窒化処理鋼板

段階で不適切なＡｌＮが形成されるので０．０６０%以下とする。

from fact that solid solution it does, content ofthe AlN many under 1280 °C, unsuitable AlN is formed with step ofthe hot rolling, it makes 0.060 % or lower.

【００１７】Ｎは前記Ａｌ、Ｓｉ等と結合して二次再結晶の安定化作用を奏させるために０．００１０%以上含有させる。しかし、その含有量が多くなると表面欠陥が生じるので０．０１３０%以下とする。Ｐは低温スラブ加熱の場合には、磁束密度を高める作用があり、この作用を奏するためには０．００５%以上必要である。一方、その含有量が多くなると冷延性が劣化するので０．０４５%以下とする。|

[0017] N connecting with aforementioned Al and Si, etcthe 0.0 010 % or higher contains in order to play stabilizing action of secondary recrystallization. But, when content becomes many, because surface defect occurs, it makesthe 0.0130 % or lower. As for P in case of low temperature slab heating, there is action whichraises magnetic flux density, in order to possess this action, it is 0.005 % or highernecessary. On one hand, when content becomes many, because cold ductilitydeteriorates, it makes 0.045 % or lower.

【００１８】Ｃｒは高磁束密度が得られるＡｌ量の範囲を拡げることを介して磁気特性を高める作用があり、そのために０．０４%以上必要である。一方、その含有量が多くなると脱炭性が劣化するので０．２５%以下とする。更に、上記元素の他にＰｂ、Ｔｅ、Ｍｏ、Ｎｂ、Ｓｅ、Ｎｉ、Ｖ、Ｓｂ、Ｂ、Ｔｉ、Ｓｎの１種類以上を適量添加すると、仕上焼鈍における鋼板の窒素の吸収、及び鋼板からの窒素の脱離を抑制し、被膜、磁性の安定性が図れると同時にそれらの元素そのものも粒界に析出することによりインヒビターを形成し、鉄損が改善される。これらの元素の添加量は０．００５%未満ではその効果が小さく、一方０．３%を超えると磁気特性上好ましくない影響を及ぼすか、あるいは意味がない。適正範囲は０．００５～０．３%である。

[0018] As for Cr through fact that range of amount of Al where thehigh magnetic flux density is acquired is expanded there is action which raises themagnetic property, it is 0.04 % or higher necessary because of that. On one hand, when content becomes many, because decarbonizing characteristicdeteriorates, it makes 0.25 % or lower. Furthermore, To other than above-mentioned element Pb , Te , Mo, Nb, Se, Ni , V, When 1 kind or more of Sb , B, Ti and Sn thesuitable amount is added, when it finishes and it controls liberation of thenitrogen from absorption, and steel sheet of nitrogen of thesteel sheet in annealing, can assure stability of coating and magnetismthe inhibitor is formed due to fact that also those element itselfprecipitate simultaneously to grain boundary, iron loss is improved. When as for addition quantity of these element under 0.005 % effect issmall, exceeds 0.3 % on one hand on magnetic property it exerts desirableinfluence, or there is not meaning. proper range is 0. 005 to 0.3 %.

【００１９】電磁鋼スラブは転炉あるいは電気炉などの溶解炉で溶製され、必要に応じて真空脱ガス処理が施され、連続鋳造または造塊－分塊圧延により製造される。電磁鋼スラブは熱間圧延に先立って加熱されるが、その加熱温度は１２８０℃未満として省エネルギーが図られる。この加熱温度では該電磁鋼スラブ中のＡｌは完全に固溶されず、不完全固溶状態となる。又さらに固溶温度の高いＭｎＳは当然ながら不完全固溶である。

[0019] Electromagnetic steel sla溶make is done with rotary furnace or electric furnace or other melting furnace, according to need vacuum degassing isadministered, is produced by continuous casting or structure lump - amountlump rolling . electromagnetic steel slab is heated preceding hot rolling,, but as for heating temperature theenergy conservation is assured as under 1280 °C. With this heating temperature as for Al in said electromagnetic steel slab solid solution it is not donecompletely, becomes imperfect solid solution state. In addition furthermore MnS where solid solution temperature is high properis imperfect solid solution.

【００２０】スラブ加熱後は熱間圧延され、必要によっては焼鈍され、あるいは焼鈍されることなく、冷間圧延される。冷間圧延は１回又は中間焼鈍を挟んで２回以上の冷間圧延を施され、最終板厚とされる。ところで本発明においては、電磁鋼スラブは１２８０℃未満の低い温度に加熱される。従って、鋼中のＡｌ、Ｍｎ、Ｓ等を不完全固溶状態としており、このままでは、鋼板中に二次再結晶を発現させるための（Ａｌ、Ｓｉ）Ｎ、ＭｎＳ等の析出型のインヒビターが存在しない。故に、二次再結

[0020] After slab heating hot rolling it is done, annealing it is d onedepending upon necessary, cold rolling it is done or without annealingbeing done. cold rolling putting between one time or intermediate annealing, is administered thecold rolling above twice, makes final plate thickness. By way regarding to this invention, electromagnetic steel slab is heated to temperaturewhose under of 1280 °C is low. Therefore, Al in steel, we designate Mn and theS etc as imperfect solid solution state, this way, (Al and Si) N in order toreveal secondary

晶発現以前に、鋼中にＮを侵入させ、インヒビターとして機能する（Ａｌ、Ｓｉ）Ｎを形成する必要がある。

recrystallization in steel sheet, inhibitor of MnS or other precipitation typedoes not exist. In reason, before secondary recrystallization revealing, invading in steel it isnecessary to form (Al and Si) N which functions, with N as the inhibitor.

【００２１】電磁鋼スラブは熱間圧延後、必要に応じて焼鈍し、冷間圧延する。冷間圧延は１回又は中間焼鈍を挟んで２回以上行われ、所定の板厚とした後、脱炭焼鈍する。脱炭焼鈍は８００～９００℃の温度で湿潤雰囲気ガス中で行うが、脱炭に引き続き（Ａｌ、Ｓｉ）Ｎ等の析出物型のインヒビターの形成を図るために、ストリップ状態でNH_3等により窒化する。窒化は６００～９００℃の温度域でドライな雰囲気（低露点）で行うのが好ましい。次いで焼鈍分離剤を塗布し、高温の仕上焼鈍を施す。

[0021] After hot rolling, according to need annealing it does el ectromagnetic steel slub, cold rolling does. cold rolling does putting between one time or intermediate annealing, after you doand specified plate thickness above twice, decarbonizing annealing. With temperature of 800 to 900 °C it does decarbonizing annealing in wetting atmosphere gas, but itcontinues to decarbonizing and in order to assure formation of theinhibitor of (Al and Si) N or other precipitate type, nitriding it does with strip condition withthe NH3 etc. As for nitriding it is desirable with temperature region of 600 to 900 °C to dowith dry atmosphere (low dew point). Next, annealing fractionating agent is applied, finished annealing of high temperature isadministered.

【００２２】仕上焼鈍過程における鋼板に吸窒、脱窒状況及び一次再結晶粒径の変化をＰｂ、Ｂ、Ｓｅ等の微量元素を添加した材料と無添加の材料について調査した。その結果を図１、図２に示す。図１に示すように、Ｂ、Ｐｂ、Ｓｅ等の元素を微量添加した鋼板は仕上焼鈍時での鋼板吸窒現象はなく、かつ高温領域迄鋼中の窒素が確保されていることが判った。一方、それらを添加していない鋼板は仕上焼鈍時の鋼板の吸窒量（追加窒化）が大きく、又窒素の抜けも大きいことが判った。

[0022] You investigated sucking/absorbing nitrogen, change of denitrification status and primary recrystallization particle diameter concerning material of material and no addition whichadd Pb , B and Se or other trace elements in steel plate in finishedannealing process. Result is shown in Figure 1 and Figure 2. As shown in Figure 1, it finished steel plate which B, the Pb and Se or other element trace addition is done and there was not a steel platesucking/absorbing nitrogen phenomenon at time of annealing, at sametime it understood to high temperature region that nitrogen in steel isguaranteed. On one hand, that it finishes steel plate which does not add those thesucking/absorbing nitrogen quantitative (Additional nitriding) of steel plate at time ofthe annealing is large, it understood in addition also coming out ofthe nitrogen is large.

【００２３】また、仕上焼鈍中の一次再結晶の粒径の成長挙動であるが、図２に示すように、Ｂ、Ｐｂ、Ｓｅ等の元素を添加したものは、一次再結晶の粒成長が抑えられているのに対し、無添加のものは、粒成長の抑止力が充分でなく、８００℃付近から早くも一次再結晶の粒成長を生じていた。Ｂ、Ｐｂ、Ｓｅを添加した材料を脱炭焼鈍後、オージェ等で分析したところ、これらは一次再結晶の粒内には存在せず、粒界に偏析していることが確認された。

[0023] In addition, it finishes and it is a growth behavior of part icle diameter of theprimary recrystallization in annealing, but as shown in Figure 2, as for those which addthe B, Pb and Se or other element, as for those of no addition,deterrent power of grain growth not to be a satisfactory, also early caused thegrain growth of primary recrystallization from 800 °C vicinity vis-a-vis grain growth of primary recrystallizationbeing held down. material which adds B, Pb and Se after thedecarbonizing annealing, when you analyzed with Auger etc, these did not existinside grain of primary recrystallization, what segregation has been done wasverified in grain boundary.

【００２４】なお、仕上焼鈍時での鋼板の吸窒（追加窒化）を抑え、かつ一次再結晶の粒成長を抑制する元素を他に調べたところ、Ｔｅ、Ｍｏ、Ｎｂ、Ｎｉ、Ｖ、Ｓｂ、Ｔｉ、Ｓｎが同様な結果を示した。以上の手段で仕上焼鈍時の吸窒（追加窒化）を抑えて一次再結晶の粒の粗大化を抑制することにより、被膜特性、磁気特性がともに良好でかつバラツキも小さい品質の優れた方向性電磁

[0024] Furthermore, it finished and held down sucking/absorbing nitrogen(Additional nitriding) of steel sheet at time of annealing, when element which at thesame time controls grain growth of primary recrystallization was inspected in otherthings, it showed result where Te , Mo, Nb,the Ni , V, Sb , Ti and Sn are similar. It finishes with means above and holds down sucking/absorbingnitrogen (Additional nitriding) at time of

鋼板が得られる。

annealing and coating characteristic and magnetic property beingsatisfactory together by controling roughening of grain of theprimary recrystallization, and directionality electromagnetic steel sheet where quality where also variation is small issuperior is acquired.

【００２５】

[0025]

【実施例】次に実施例について述べる。表１に示す成分組成のスラブを表２で示す条件で加熱し、２．３mmの厚みに熱間圧延し、熱延板を冷間圧延し、０．３mmの板厚とした。その後に、８３０℃＋１５０秒、H_2 ７５%、N_2 ２５%のガスに加湿して露点約６０℃の雰囲気ガスに調整した条件下で脱炭焼鈍し、続いてNH_3により鋼板を窒化し、窒素量としては１８０～２００ppm とした。

[Working Example(s)] Next you express concerning Working E xample. It heated with condition which shows slab of component composition which isshown in Table 1 with Table 2 hot rolling did in thickness of the2.3 mm, cold rolling did hot rolled plate, made plate thickness of 0.3 mm. after that, humidifying gas of 830 °C + 150 second , H2 75 % and N2 25 %, decarbonizing annealing it did under condition which you adjusted theatmosphere gas of dew point approximately 60 °C nitriding it did steel sheetcontinuously with NH3 , it made 180 to 200 ppm as nitrogen amount.

【００２６】次いでＭｇＯを主成分とする焼鈍分離剤を鋼板に塗布し、コイルに巻き取った後、H_2 ７５%、N_2 ２５%の雰囲気で仕上焼鈍を１２００℃×２０時間行った。得られた方向性電磁鋼板の磁気特性、被膜特性を測定し、その結果を表３に示す。

[0026] Next, it applied annealing fractionating agent which desi gnates MgO as main component tothe steel sheet, after retracting in coil, it finished with atmosphereof H2 75 % and N2 25 % and 1200 °C X 2 0 hour did annealing. magnetic property of directionality electromagnetic steel sheet which is acquired, coating characteristic ismeasured, result is shown in Table 3.

【００２７】

[0027]

【表１】 [Table 1]

※	符号	鋼成分 (%)								
		C	Si	Mn	S	Al	N	P	Cr	その他の成分
	1	0.050	3.26	0.134	0.007	0.0284	0.0074	0.025	0.05	Sb 0.001
	2	0.065	3.24	0.136	0.008	0.0271	0.0076	0.031	0.07	Se 0.003
○	3	0.054	3.25	0.150	0.006	0.0243	0.0073	0.018	0.14	Pb 0.03
○	4	0.050	3.21	0.129	0.006	0.0287	0.0075	0.022	0.20	B 0.05
○	5	0.053	3.23	0.138	0.008	0.0265	0.0072	0.034	0.11	Se 0.03
○	6	0.061	3.24	0.140	0.009	0.0275	0.0079	0.011	0.07	Te 0.05 Nb 0.007
○	7	0.066	3.21	0.136	0.008	0.0271	0.0076	0.029	0.06	Sb 0.06 Ti 0.09
○	8	0.057	3.28	0.154	0.006	0.0241	0.0079	0.007	0.09	Sn 0.05 Ni 0.01
○	9	0.051	3.20	0.139	0.005	0.0247	0.0071	0.026	0.15	Mo 0.1 V 0.02
○	10	0.059	3.25	0.148	0.007	0.0275	0.0077	0.019	0.08	Pb 0.05 B 0.01

※ ○印は本発明例

【００２８】 [0028]

【表２】 [Table 2]

（表１のつづき）

※	符号	スラブ加熱温度（℃）	脱炭時の露点（℃）	鋼板窒素量（ppm）	仕上焼鈍時の雰囲気ガス種類（%）
	1	1200	61	187	H_2 75 N_2 25
	2	1220	59	175	H_2 75 N_2 25
○	3	1190	60	181	H_2 75 N_2 25
○	4	1230	61	201	H_2 75 N_2 25
○	5	1160	59	185	H_2 75 N_2 25
○	6	1225	62	197	H_2 75 N_2 25
○	7	1145	59	191	H_2 75 N_2 25
○	8	1190	60	185	H_2 75 N_2 25
○	9	1230	61	194	H_2 75 N_2 25
○	10	1150	59	183	H_2 75 N_2 25

※ ○印は本発明例

【００２９】 [0029]

【表３】 [Table 3]

※	符号	磁束密度 B_8 (T)	鉄損 $W_{17/50}$ (W/kg)	被膜性状 (被膜欠陥状況)
	1	1．９１	1．００	シモフリ発生　大
	2	1．９２	0．９８	シモフリ発生　中
○	3	1．９２	0．９８	良　好
○	4	1．９３	0．９７	良　好
○	5	1．９２	0．９８	良　好
○	6	1．９３	0．９８	良　好
○	7	1．９３	0．９７	良　好
○	8	1．９２	0．９８	良　好
○	9	1．９２	0．９９	良　好
○	10	1．９３	0．９７	良　好

※　○印は本発明例

【００３０】

【発明の効果】本発明によれば、実施例にみられるように仕上焼鈍過程での追加窒化がないために、被膜欠損の発生が皆無であり、かつ微量元素が粒界に偏析して、インヒビター機能をもつために高温まで一次再結晶粒径が保持され、その結果として、方位のそろった二次再結晶が得られ、極めて磁気特性の優れた方向性電磁鋼板が製造され得る。

[0030]

[Effects of the Invention] In this invention we depend, As seen in Working Example, it finishes and because there is not anadditional nitriding with annealing process, occurrence of coating deletion is nil, atthe same time trace elements segregation does in grain boundary, because it has inhibitor function primary recrystallization particle diameter is kept to high temperature, secondary recrystallization where the azimuth is even as result, is acquired, directionality electromagnetic steel sheet where quitemagnetic property is superior can be produced.

【図面の簡単な説明】

【図１】仕上焼鈍過程における鋼中の窒素量の変化を示す図である。

【図２】仕上焼鈍過程における一次再結晶の変化を示す図である。

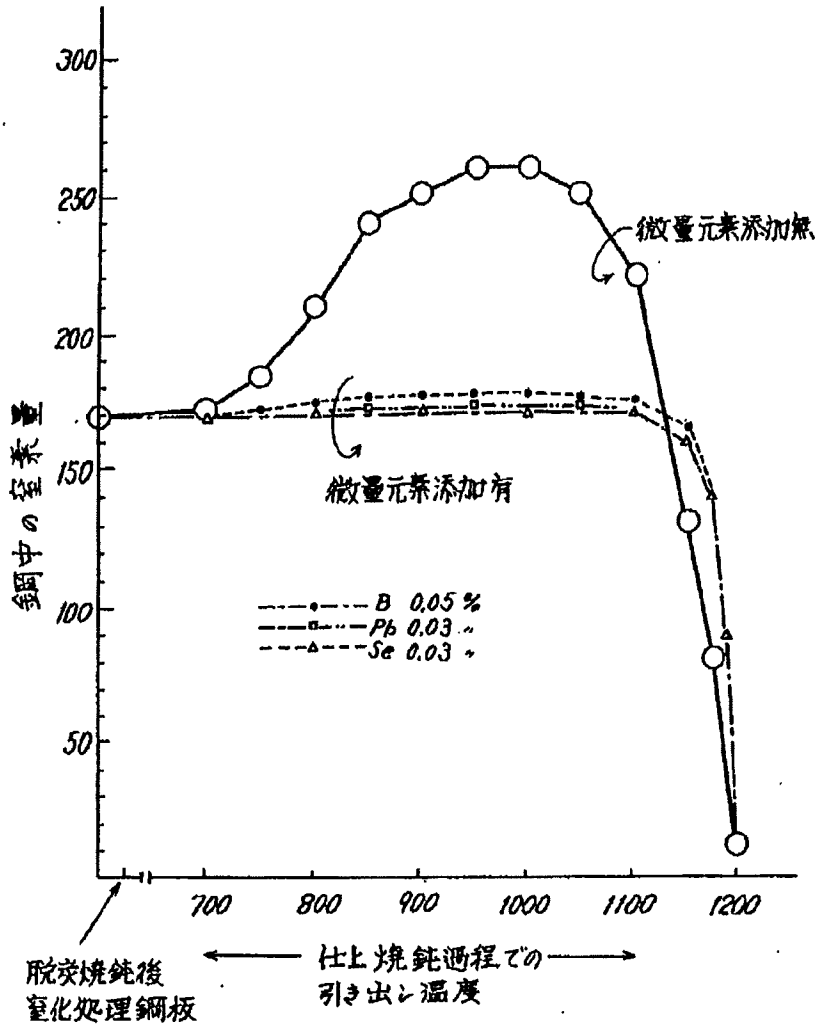
[Brief Explanation of the Drawing(s)]

[Figure 1] It is a figure which shows change of nitrogen amount i n steel in thefinished annealing process.

[Figure 2] It is a figure which shows change of primary recrystall ization in finishedannealing process.

【図１】

[Figure 1]


300
250
200
150
100
50
鋼中のS含有量
微量元素添加無
微量元素添加有
B 0.05 %
Pb 0.03 〃
Se 0.03 〃
700
800
900
1000
1100
1200
脱炭焼鈍後
窒化処理鋼板
仕上焼鈍過程での
引き出し温度